HITACHI 4500-Il INSTRUCTIONS

The following instructions provide a guide to performing simple tasks on the microscope. Initial sections (A to D) describe how to insert a sample and obtain an image. Subsequent sections provide information on more COni~!CX operations such as changing the accelerating voltage or the detecton Sections K and L provide infonnation on what to do at the end of a session.

Compared with the JEOL 840 SEM there are more variable parameters available to the operator. Optimum imaging conditions will vary from user to user depending on their samples. It is advisable that you spend some time attempting to determine which conditions are best for your sample.

When using this microscope please note the following rules:

RULE 1: DON'T TOUCH A CONTROL IF YOU ARE NOT SURE OF THE OUTCOME OF THAT ACTION.
RULE 2: NEVER, EVER FORCE ANYTHING BEYOND FINGER STRENGTH
RULE 3: IF IN DOUBT; ASK FOR HELP

A. START-UP

1. Turn on the visual display by turning toggle switch **DISPLAY POWER** to 1. (These toggle switches must be pulled out slightly before switching - DO NOT FORCE THEM)
2. Turn the toggle switch SC **AIRLOCK VALVE** from **CLOSED** to **AUTO** (OPEN)- a few seconds later the green light on the HV off/ready display will come on. If it doesn't please contact EM staff for help.

B. INSERT SAMPLE

1. Put specimen stub onto specimen holder> inc exact mechanism will vary depending upon the specimen type. EM Unit staff will advise the best method for your sample when you art being trained. The specimen holder and stub MUST be set so that so that thtt top of the specimen is level with or below tIze gauge.
2. Ensure toggle switch on SC **AIRLOCK VALVE DISPLAY** is set to S.E.C.
3. Press **AIR** on **Sc AIRLOCK VALVE DISPLAY -** this will bleed air into the specimen exchange chamber. It will take 2~3O seconds for the chamber to come to air.
4. Ensure stage settings **(X,Y,Z,R** and T) have been return to their default positions (displayed on the specimen chamber).
5. Open specimen exchange chamber door Do not force.
6. Push specimen exchange rod forward slightly so it protrudes through the specimen exchange chamber door and screw specimen holder on to screw thread of the specimen exchange rod. **PLEASE MAKE SURE YOU DO NOT GET** A **CROSS THREAD.** Pull specimen exchange rod back into specimen exchange chamber door.
7. Close airlock door

8. Press **EVAC** on **SC AIRLOCK VALVE DISPLAY** - this will pump the specimen exchange chamber.
9. Observe SEC vacuum display as status lights show vacuum goes from AIR to LOW to HIGH.
10. When SEC vacuum is FilCH (green light is on), open airlock door, by turning airlock level anticlockwise about one half turn. **DO NOT FORCE THIS LEVER,** it requires minimal effort to open.
11. When airlock door is open, fully insert specimen exchange rod into specimen chamber. Watch the specimen being inserted and ensure that it sits properly inside the specimen chamber. When rod is fully inserted, unscrew rod from sample by turning rod anticlockwise.
12. When rod is unscrewed, withdraw the rod into the specimen exchange chamber.
13. Close airlock door by turning airlock level clockwise about one half turn.

C TURNING THE BEAM ON – *GETTING AN IMAGE*

1. Select the required accelerating voltage Press **PF1** Vacc
2. Using numeric keyboard key in desired accelerating voltage e.g. **5kv.**
3. Press **ENTER**
4. Microscope will automatically go to 5kV.
5. Press **PF16 EXIT** to clear screen
6. Press the ON bunon on the HV display (middle button). The red left hand button will flash while the high tension comes on. When the beam current has been reached the light will stop flashing.
7. Set magnification to IQOOX. You can do this by manually turning the **MAGNIFICATION** knob or pressing the **PRESET** switch on the magnification display.
8. Locate area of interest using X and V stage shifts (you may need to go to low magnification see section I).
9. Turn the **COARSE** FOCUS knob until a nominally focused image is obtained.
10. Press **ABC** button on the image adjust section to automatically correct brightness and contrast.#
11. Press **SMALL AREA DISPLAY** button on scan speed section.
12. Increase magnification to -10,000 times by manually turning the **MAGNIFICATION** knob.
13. Focus using FINE **FOCUS knob**
14. Refine sharpness of image with STIGMA/ALIGNMENT X AND Y knobs
15. Return to **TV** or SLOW SCAN **MODES** on scan speed section.
16. If you wish to move the sample to a different area, manipulate the specimen with the X AND Y shifts on the specimen chamber. You can also adjust the height, **Z** tilt, T and rotation, R of the sample. If you adjust the tilt or height of the sample, you will need to consider the effects of that movement with respect to any detectors inserted into the chamber. Always return **X , Y, Z** , R and T controls to their preset levels before removing a sample.
11. When you have moved to a new area you may need to repeat steps 9-16.

The ABC control provides 'ideal brightness and contrast levels for photography. But may not always provide the levels of brightness and contrast you desire. An alternative method is to use the BRIGHTNESS and CONTRAST up/down arrowed keys on the image adjust section.

D. TAKING A PHOTOGRAPH

1. Obtain the image you desire.
2. Press **ABC** button on the image adjust section to automatically alter brightness and contrast to the correct levels for the camera.
3. Press **DIRECT** switch on the photographic control section.
4. Wait for the line scan to perform on cycle and expose the film. Exposure is complete when the light on the **DIRECT** switch goes off.
5. Withdraw the film from the cassette.

WHAT TO DO WHEN IT ALL GOES DARK - DON'T PANIC: DON'T FIDDLE WITH ANYTHING

1. ASK THE EM UNIT STAFF FOR HELP
2. **READ THE MANUAUL INSTRUCTIONS**
3. **ASK THE EM UNIT STAFF FOR HELP**

E. CHANGING ACCELERATING VOLTAGE

Unlike conventional SEMs, the 4500 is able to operate at a range of accelerating voltages, from 1-30kV. Lower accelerating voltages may provide more surface sensitive information and reduce the size of the electron-beam specimen interaction volume.

I. Press FEll Vace
2. Using numeric keyboard key in desired accelerating voltage e.g. 5kV.
3. Press **ENTER**
4. Microscope will automatically go to 5kV.
5. Press **PF16 EXIT to** clear screen
6. Refocus image as described above
7. Go to ALIGN COLUMN - section F

F. TO ALIGN THE COLUMN

1. Obtain a focused image at about 510,000 times magnification.
2. Press PF3 COLUMN ADJUST
3. With $ sign against **BEAM ALIGN** Press ENTER, an aperture will be inserted which is in view on the screen
4. Increase magnification so aperture fills screen.
5. Centre aperture on screen with **ALIGNMENT X and Y** knobs.
6. Hit ENTER to move $ sign against **APERT ALIGN** Press ENTER again
7. The image will oscillate on the screen, minimise oscillation with **ALIGNMENT X and Y** knobs.
8. Hit ENTER to move $ sign against STIG ALIGN X Press ENTER again
9. The image ~l oscillate on the screen, minimise oscillation with **ALIGNMENT X and Y** knobs.

10. Hit ENTER to move $ sign against **STIG ALIGN Y** Press **ENTER again**
11. The image will oscillate on the screen, minimise oscillation with **ALIGNMENT X and Y** knobs.
12. Press PF16 EXIT to finish.

G. CHANGING THE DETECTOR

The 4500 has two secondary electron detectors, an upper detector which sits above the specimen and a lower detector which sits to the side of the specimen. Each detector provides a different perspective of the specimen. To change the detector:

1. Press **PF2 COLUMN COND** 1.
2. Using up/down arrowed keys on keyboard move cursor $ down to line 8. This will tell you which detector is currenfly in use, e.g. 'Upper'.
3. Press left/right arrowed keys on keyboard. Below the menu the INPUT detector, i.e. the one to be chosen will be displayed. In this case 'Lower.
4. Press ENTER and the detector displayed at INPUT will become active.
5. Press **PF16 EXIT** to finish.
6. Changing detectors may require you refocus the image and adjust brightness and contrast.
7. n.b. The upper detector generally works better at short working distances <18. The lower detector generally works better at long working distances >18. See figure below. You may accordingly need to change the specimen height.

H. CHANGING THE WORKING DISTANCE

1. The working distance is changed by using the **Z** adjust on the specimen chamber. The approximate working distance can be read directly from this control.
2. Changing the working distance will defocus the image. Focus can be obtained using the COARSE and FINE focus controls.
3. Coarse focus can be readjusted by pressing PF2 COLUMN COND 1.
4. Using up/down arrowed keys on keyboard move cursor -> down to line 3.
5. Enter desired working distance using numeric keyboard keys e.g. 20.
6. Press ENTER
7. Microscope will automatically go to a working distance of 20.
8. Press **PF16 EXIT** to clear screen.

I. LOW MAGNIFICATION MODE

Often it can be difficult to locate the area you want on a specimen. In this case use Low Magnification Imaging, here the lowest magnification is x20.

1. Press **LOW MAG** switch on display panel (the green light in the switch will glow). This will automatically set the magnification to x500.
2. Alter magnification using **MAGNIFICATION** knob to required level. Using **X and V** controls to obtain area of interest.
3. Turn off low magnification mode by pressing **LOW MAG** (the green light in the switch will dim).

J. AUTOMATIC FOCUS (AFC)

If you prefer the microscope will automatically focus the image. It is, however, less reliable than manual focus, and requires that the image prior to focusing contains some contrast for the AFC to operate.

1. To coarse focus press the **SEARCH button** on the focus panel. The image will disappear and the monitor line scan will appear.
2. Press the **AUTO button** on the focus panel. This will coarsely focus the image.
3. To fine focus only press the **AUTO** button on the focus panel. This will fine focus the image.

K. REMOVING A SPECIMEN

1. Press **HV OFF button. Electron** emission will cease.
2. Set magnification to -1OOOX. You can do this by manually turning the **MAGNIFICATION** knob or pressing the PRESET switch on the magnification display.
3. Return **X,Y,Z,R** and T controls to their preset levels - listed on specimen chamber.
4. Open airlock door, by turning airlock level anticlockwise about one half turn. **DO NOT FORCE THIS LEVER,** it requires minimal effort to open.
5. When airlock door is open, fully insert specimen exchange rod into specimen chamber. When rod is fully inserted, screw rod onto specimen holder by turning rod clockwise.
6. When rod is screwed onto the holder, withdraw the rod into the specimen exchange chamber. Watch to ensure the specimen is attached to rod.
7. Close airlock door by turning airlock level clockwise about one half turn.
8. Ensure toggle switch on **SC AIRLOCK VALVE DISPLAY** is set to S - E. C.
9. Press **AIR on SC AIRLOCK VA.LVE DISPLAY** - this will bleed air into the specimen exchange chamber. This takes about 20-30 seconds.
10. Open specimen exchange chamber door. Do not force.
11. Push specimen exchange rod forward slightly so it protrudes through the specimen exchange chamber door and unscrew specimen holder from the specimen exchange rod. Pull specimen exchange rod back into specimen exchange chamber door.
12. Close airlock door
13. Press EVAC on SC AIRLOCK VALVE DISPLAY - this will pump the specimen exchange chamber.
14. Observe SEC vacuum display as status lights show vacuum goes from AIR to LOW to HIGH.

L. FINISHING A SESSION

1. Turn the **SC AIRLOCK VALVE** from **AUTO (OPEN**) to **CLOSED** - the green light on the HV off/ready display will turn off..
2. Turn off the visual display by turning **DISPLAY POWER** to **O.**
3. Fill in log book.